Interlaboratory Study 2000-1
Toxaphene Standards and
Air Sample Extract

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1. INTRODUCTION

Toxaphene has been used as a pesticide throughout the world, and its residues are found in all environmental matrices. Technical toxaphene is a mixture of chlorinated camphene derivatives, primarily bornanes ($C_{10}H_{18-n}Cl_n$) and bornenes ($C_{10}H_{16-n}Cl_n$), with a range of 6 to 10 chlorine atoms. This results in the possibility of over 600 individual components, known as toxaphiene congeners. Not all of the possible congeners have been identified, but many congeners of environmental significance have been identified by Parlar numbers(1). However, the residues in the environment change due to weathering, bioaccumulation and degradation, so that quantitative analysis of toxaphene can be difficult(2).

Interlaboratory Study 2000-1 was initiated as a cooperative project between the Meteorological Service Canada (MSC) of Environment Canada (EC) and Laboratory Services Branch of the Ontario Ministry of the Environment (LSB-MOE) to assess the current performance of environmental laboratories analyzing toxaphene in air. Any problems identified by the study were intended to help improve the between-laboratory comparability for this type of analysis.

2. STUDY DESIGN

2.1. Source of Materials

A concentrated mixture of 6 toxaphene congeners was purchased from Ehrenstorfer, Germany. It was diluted by a factor of 30 in iso-octane. 2 mL aliquots were sealed in amber ampoules and labeled Sample TOX2000-1-1.

A concentrated solution of toxaphene Parlar 42 was purchased from Axact Standards. It was diluted by a factor of 30 in iso-octane. 2 mL aliquots were sealed in amber ampoules and labeled Sample TOX2000-1-2.

A technical toxaphene mixture was purchased from Restek. It was diluted by a factor of 1000 in iso-octane. 2 mL aliquots were sealed in amber ampoules and labeled TOX2000-1-3.

Large-volume air samples were collected during the summer of 1998 is southern Alabama, U.S.A., using a glass fibre filter - polyurethane foam (PUF) trap. The filters and PUFs were extracted by Dr. Bidleman's laboratory at MSC and the extracts were pooled together. 5 mL aliquots of the extract were sealed in amber ampoules and labeled TOX2000-1-4.

2.2 Sample Distribution

Over 15 environmental laboratories were invited to participate in Canada, the U.S.A., and Europe. Eleven laboratories agreed to participate. All participants were assigned an identification code.

Sample sets, consisting of the four different ampoules, were sent by courier to the participants on April 4, 2000. An instruction sheet and report form was included with the samples, as well as a methodology questionnaire. Examples are included in Appendix 5. Electronic versions of the report form and methodology questionnaire were provided by e-mail to all of the participants.

The concentration of the toxaphene Parlars in samples TOX2000-1-1 and TOX2000-1-2 were provided to the participants on the instruction sheet. They were provided to the participants to use as calibration standards. Samples TOX2000-1-3 and TOX2000-1-4 were "unknowns", and the participants were asked to analyze the solutions using their routine methods and report their results.

3. STUDY RESULTS

Results were received from all 11 participants. Two participants did not use the solutions of individual Parlars provided, and only reported a Total Toxaphene result for TOX2000-1-3 and TOX2000-1-4. Three participants provided some additional results using their own in-house calibration solutions or a duplicate result (Appendix 1, Table 2).

A preliminary table of results was distributed to the participants on November 24, 2000. Two participants reviewed their work and provided revised data. The final data set is provided in Appendix 1, Table 1.

An interlaboratory mean, standard deviation, and relative standard deviation (%RSD) was calculated for the data set and is included in Appendix 1, Table 3. No outliers were removed.

The target value for Total Toxaphene is provided for sample TOX2000-1-3. The percent recovery for each participant is provided in Appendix 1, Table 4.

To provide a rapid assessment of between-laboratory performance, a bar graph was prepared for each Parlar and for the Total Toxaphene result reported in samples TOX2000-1-3 and TOX2000-1-4. These graphs are in Appendix 2. Because some of the participants reported a sum of Parlars 40 and 41, and others reported the two Parlars individually, only one graph was prepared for the sum of Parlars 40 and 41. The target value for Total Toxaphene in TOX2000-1-3 is also indicated in Figure 1.

The responses to the methodology questionnaire are summarized in Appendix 3, Table 4. As much of the information is confidential, most of the information is summarized as to the number of laboratories using the same type of equipment, solvents, standards, etc. The only exception was the response to the question regarding the calculation of "Total Toxaphene".

4. DISCUSSION

Sample TOX2000-1-3 and TOX2000-1-4 did not have "known" values for the amount of individual toxaphene Parlars present. The results in Table 1 show a broad range of reported values. With no outliers removed, the %RSD for the Total Toxaphene and the individual Parlars targeted for this study ranged from 34 to 65%, except for Parlar 62 in Sample TOX2000-1-4. In the case of Parlar 62 in TOX2000-1-4, the result from Laboratory 2K102 is significantly different from the other participants. Laboratory 2K102 may have made a calculation or transcription error for this parameter, that was not investigated when the preliminary table of results was distributed to the participants. All of the results for the individual toxaphene Parlars are assumed to have been calculated with the study solutions TOX2000-1-1 and TOX2000-1-2 as the calibration standards.

Laboratory 2K110 reported higher results than the other participants for most of the parameters in sample TOX2000-1-3 (Figures 1 to 7). However in sample TOX2000-1-4 their results were more consistent with the other participants (Figures 8 to 14). They did not indicate that they handled the two samples differently.

The target value for Total Toxaphene in Sample TOX2000-1-3 is provided in Appendix 1, Table 4), as well as the percent recovery for each participant. While the ILS mean recovery is close to the target (112%), individual results range from 46% to 181% of the target. There does not appear to be a correlation between low recovery and the range of homologue groups summed to calculate "Total" Toxaphene. Laboratory 2K108 (46%) sums over the range of 6 to 10 chlorine atoms, while Laboratory 2K110 (181%) only sums over the range of 6 to 9 chlorine atoms. A detailed comparison of the chromatograms from the participants, reviewing the number of individual peaks that are integrated and summed for each homologue group, would be required to identify the differences in recovery of Total Toxaphene in this sample.

The participants in this study have several differences in their methodology that are probable contributors to the range of the results (Appendix 3, Table 4). While most laboratories are using the same type of instrumentation (8 out of 11 are using gas chromatography-electron capture negative ion detection (GC/ECNI)), they are almost all using different gas chromatography (GC) columns and different temperature programs. These are two significant areas of the methodology that can affect the elution order of the many toxaphene components, there-by affecting which peaks are integrated and summed. As well, a variety of different labeled standards are also used. Not all participants correct

for recovery. Some participants correct for chlordane and endosulfan interferences. These are all contributing factors leading to quantitation differences between the participating laboratories.

Laboratory 2K111 noted that there was a slight retention time difference between the technical toxaphene peak assigned as Parlar 44 (based on published literature) and that of the closest candidate peak in the solution TOX2000-1-1. They reported two sets of values for this parameter (Tables 1 and 2). Laboratory 2K104 also reported results using their own calibration standards as well as those supplied for the study. For most of the results, the two sets of samples have close agreement. However, the largest difference is found between the Parlar 44 results using the different calibration standards. Laboratory 2K108 reported duplicate results for TOX2000-1-4, demonstrating good within-laboratory precision, as the relative percent difference between the results was 3% or less.

At the present time there are no reference standards or certified reference materials (CRMs) for toxaphene in air. The development of such materials would aid in improving the consensus in reporting data for this type of analysis(3).

As noted above, the participating laboratories have a variety of techniques for calculating "Total" toxaphene. Four of the participants indicated that they subtract chlordane and endosulfan interferences. Different ions are monitored by the participants, and the range of toxaphene homologues summed are different. While Laboratory 2K105 appears to have the smallest range summed (Table 4), they do not have the lowest "Total" results (Figures 1 and 8). The variability of these results and techniques suggest that a standardized approach (i.e. a "standard method") would reduce between-laboratory variability.

5. CONCLUSION

The results from this interlaboratory study demonstrate a spread of approximately ±50% between the participants. Differences in methodology such as GC column, temperature program, and labeled standards appear to be important sources of the between-laboratory variability. Variations in the calculation technique for "Total" toxaphene also contribute to a lack of consensus between the participants. The need for reference standards and CRMs, as well as a "standard method" for toxaphene analysis, would help reduce the between-laboratory differences.

6. BIBLIOGRAPHY

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TABLE 1 - PARTICIPANTS' RESULTS

PARAMETER	2K101	2K102	2K103	2K104	2K105	2K106	2K107	2K108	2K109	2K110	2K111
SAMPLE TOX2000-1-3	pg/µL	ng/mL	pg/µL	pg/µL	pg/µL	ng/mL	ng/mL	pg/µL	ng/mL	pg/µL	pg/µL
Total Toxaphene	133.93 +/ 7.81	117.44	133	not determined	177	215 ± 43 (n = 2)	250	64.535	112	256	131
Toxaphene Parlar No. 26	0.50 +/ 0.09	0.36		0.54	0.455		1.07	0.864	0.44	1.56	0.751
Toxaphene Parlar No. 40	1.27 +/ 0.22	*** **2.20**		1.21	1.760		*** **0.917**	1.943 *	1.55	* **3.93**	0.912
Toxaphene Parlar No. 41	0.34 +/ 0.07			0.25	0.49				0.54		
Toxaphene Parlar No. 42	3.98 +/ 0.64	3.98		3.89	3.70		4.725	5.642	3.99	10.7	5.17
Toxaphene Parlar No. 44	6.05 +/ 0.99	3.42		4.79	0.495		3.68	4.253	1.84	6.09	<1.5 (note 1)
Toxaphene Parlar No. 50	1.52 +/ 0.11	1.44		1.38	1.75		1.83	1.649	1.34	3.29	1.52
Toxaphene Parlar No. 62	10.98 +/ 1.25	9.79		3.59	3.13		18	8.520	4.23	14	5.31
SAMPLE TOX2000-1-4											
Total Toxaphene	19.06 +/ 1.11	44.22	26.3	not determined	37.2	89.2 ± 5 (n = 3)	21.9	9.504	22.3	27.5	44.3
Toxaphene Parlar No. 26	0.21 +/ 0.05	0.2		0.11	0.530		0.505	0.314	0.31	0.242	0.477
Toxaphene Parlar No. 40	0.13 +/ 0.01	*** **0.30**		0.37	0.459		*** **0.249**	0.556 *	0.45	* **0.602**	0.405
Toxaphene Parlar No. 41	0.089 +/ 0.001			0.12	0.011				0.13		
Toxaphene Parlar No. 42	0.42 +/ 0.04	0.074		0.36	0.627		0.478	0.705	0.46	0.666	0.621
Toxaphene Parlar No. 44	2.06 +/ 0.06	0.99		2.01	0.323		1.64	1.974	0.91	1.98	<0.7
Toxaphene Parlar No. 50	0.28 +/ 0.03	0.122		0.44	0.484		0.543	0.473	0.39	0.105	0.565
Toxaphene Parlar No. 62	1.18 +/ 0.06	11.53		0.07	0.238		ND	ND	0.2	0.606	<0.8
		***** 40 and 41 combined**					***** 40 and 41 combined**	* 40 & 41		*** 40 and 41 combined**	Note 1: conc. based on MOE cal std.

TABLE 2 - ADDITIONAL RESULTS

PARAMETER	2K104A	2K111A	2K108D
SAMPLE TOX2000-1-3	pg/µL	pg/µL	pg/µL
Total Toxaphene			
Toxaphene Parlar No. 26	0.60		
Toxaphene Parlar No. 40	* 1.61		
Toxaphene Parlar No. 41			
Toxaphene Parlar No. 42	3.93		
Toxaphene Parlar No. 44	3.30	7.91 (note 2)	
Toxaphene Parlar No. 50	1.38		
Toxaphene Parlar No. 62	3.02		
SAMPLE TOX2000-1-4			
Total Toxaphene			9.65
Toxaphene Parlar No. 26	0.11 i		0.341
Toxaphene Parlar No. 40	* 0.5		0.496 *
Toxaphene Parlar No. 41			
Toxaphene Parlar No. 42	0.37		0.640
Toxaphene Parlar No. 44	1.52	3.76 (note 2)	1.696
Toxaphene Parlar No. 50	0.44		0.47
Toxaphene Parlar No. 62	0.06 i		ND
	i: with interference * 40 & 41 Quantified against our quant. standard	Note 2: conc. based on technical toxaphene	* 40 & 41

TABLE 3 - STATISTICAL CALCULATIONS
RESULTS IN pg/µL

PARAMETER	2K101	2K102	2K103	2K104	2K105	2K106	2K107	2K108	2K109	2K110	2K111	MEAN	STD DEV	n	%RSD
SAMPLE TOX2000-1-3															
Total Toxaphene	133.93	117.44	133		177	215	250	64.535	112	256	131	158.99	63.332	10	39.83%
Toxaphene Parlar No. 26	0.5	0.36		0.54	0.455		1.07	0.864	0.44	1.56	0.751	0.73	0.389	9	53.49%
Toxaphene Parlar No. 40&41	1.61	2.2		1.46	2 250		0.917	1.943	2.09	3.93	0.912	1.92	0.907	9	47.17%
Toxaphene Parlar No. 42	3.98	3.98		3.89	3.70		4.725	5.642	3.99	10.7	5.17	5.09	2.207	9	43.38%
Toxaphene Parlar No 44	6.05	3.42		4.79	0.495		3.68	4.253	1.84	6.09	<1.5	3.57	1.976	9	55.36%
Toxaphene Parlar No. 50	1.52	1.44		1.38	1 75		1.83	1.649	1.34	3.29	1 52	1.75	0.602	9	34.44%
Toxaphene Parlar No. 62	10.98	9.79		3.59	3.13		18	8.520	4 23	. 14	5.31	8.62	5.118	9	59.40%
SAMPLE TOX2000-1-4															
Total Toxaphene	19.06	44.22	26.3		37.2	89.2	21.9	9.504	22.3	27.5	44.3	34.15	22.300	10	65.30%
Toxaphene Parlar No. 26	0.21	0.2		0.11	0.530		0.505	0.314	0.31	0.242	0.477	0.32	0.150	9	46.55%
Toxaphene Parlar No. 40&41	0.219	0.3		0.49	0.470		0.249	0.556	0.58	0.602	0.405	0.43	0.145	9	33.69%
Toxaphene Parlar No. 42	0.42	0.074		0.36	0.627		0.478	0.705	0.46	0.666	0.621	0.49	0.197	9	40.10%
Toxaphene Parlar No. 44	2.06	0.99		2.01	0.323		1.64	1.974	0.91	1.98	<0.7	1.40	0.670	9	47.89%
Toxaphene Parlar No. 50	0.28	0.122		0.44	0.484		0.543	0.473	0.39	0.105	0.565	0.38	0.172	9	45.45%
Toxaphene Parlar No. 62	1.18	11.53		0.07	0.238		ND	ND	0.2	0.606	<0.8	2.09	4.181	9	200.14%

TABLE 4 - PERCENT RECOVERY OF TOTAL TOXAPHENE SAMPLE TOX2000-1-3

	Total Toxaphene	% Recovery
TARGET pg/µL	141	
2K101	133.93	94.99%
2K102	117.44	83.29%
2K103	133	94.33%
2K105	177	125.53%
2K106	215	152.48%
2K107	250	177.31%
2K108	64.535	45.77%
2K109	112	79.43%
2K110	256	181.56%
2K111	131	92.91%
MEAN	158.99	112.76%
STD DEV	63.332	42.611%
n	10	10

8. APPENDIX 2 - GRAPHS

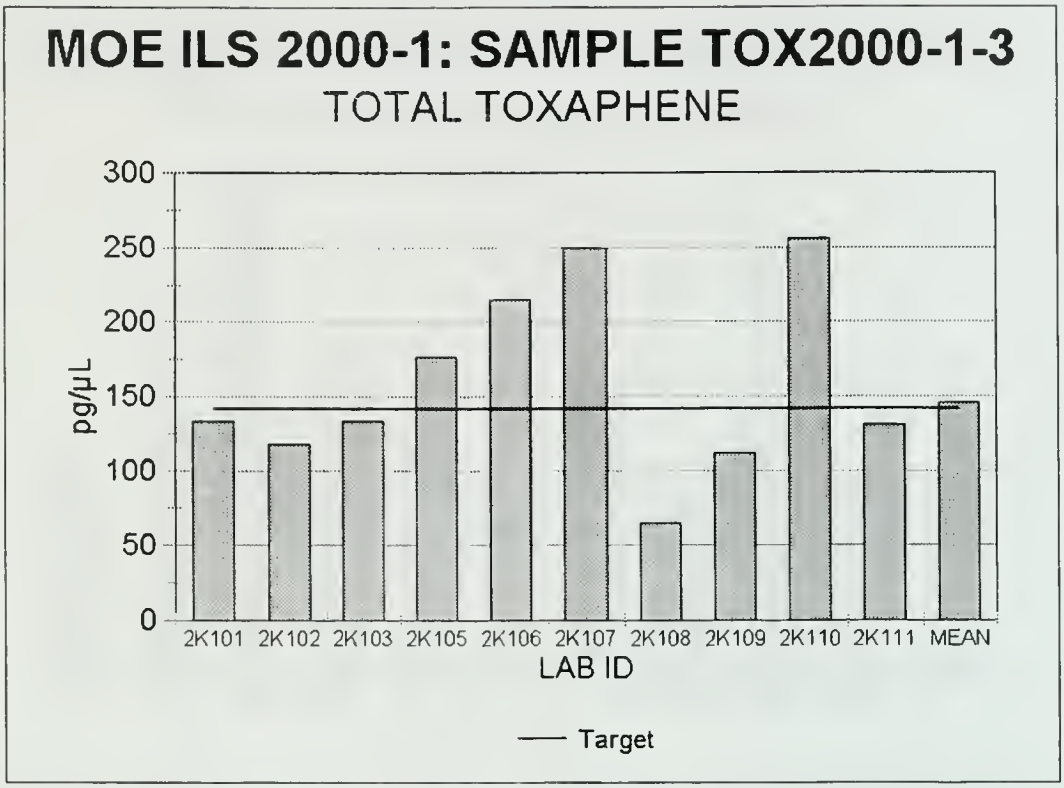

Figure 1 - Total Toxaphene in Sample TOX2000-1-3

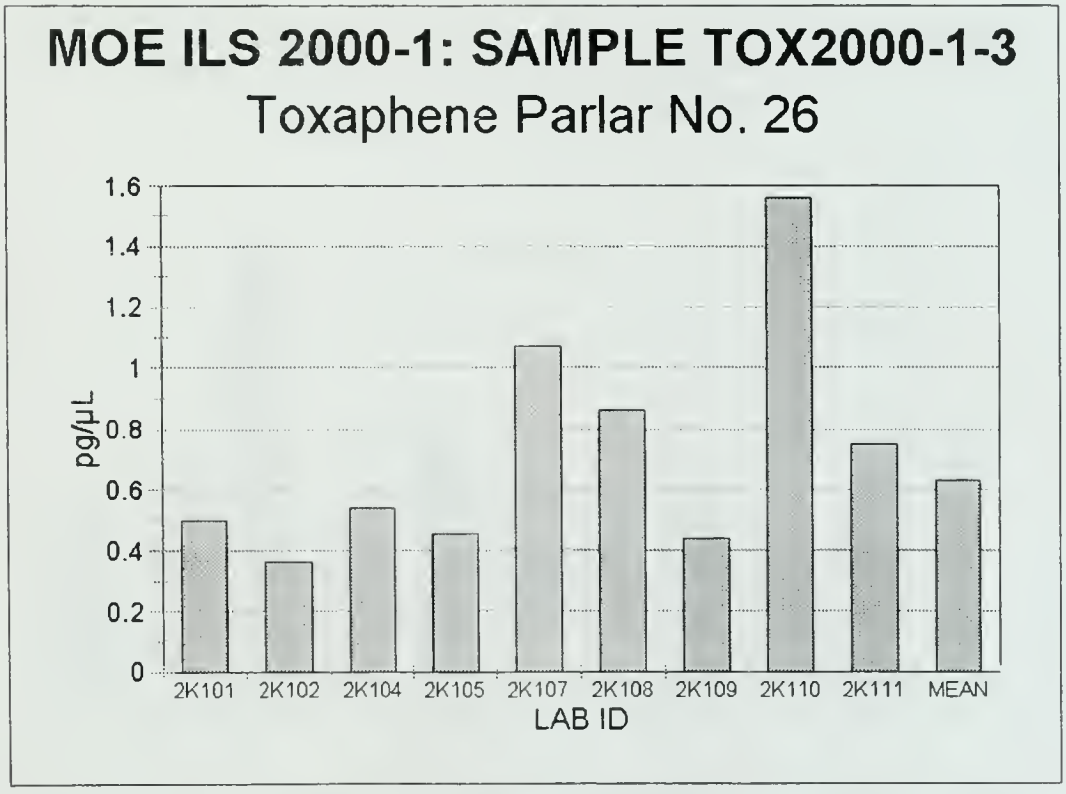

Figure 2 - Parlar 26 in Sample TOX2000-1-3

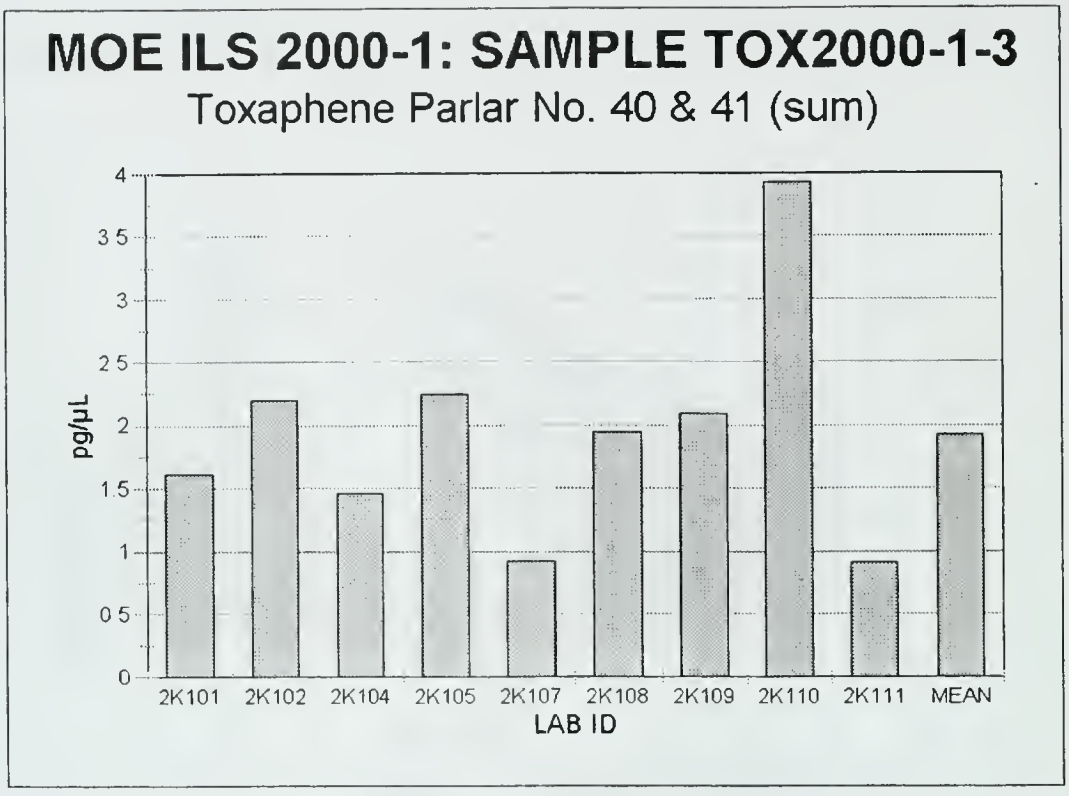

Figure 3 - Sum of Parlars 40 & 41 in Sample TOX2000-1-3

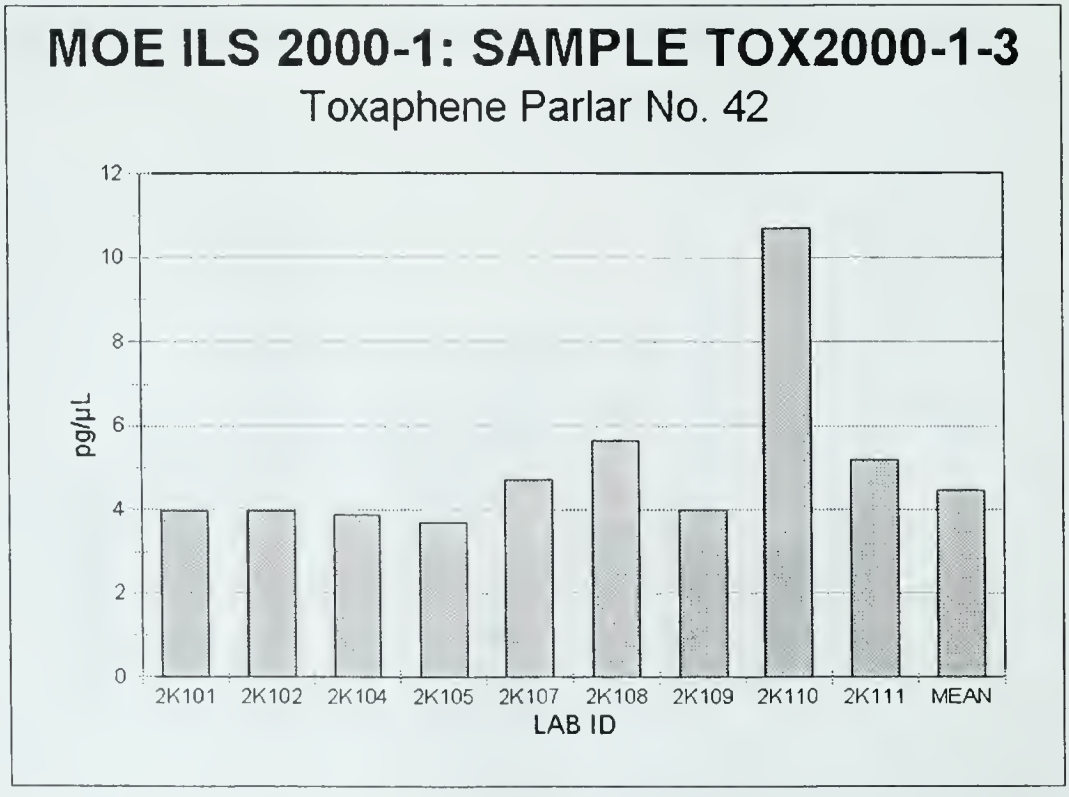

Figure 4 - Parlar 42 in Sample TOX2000-1-3

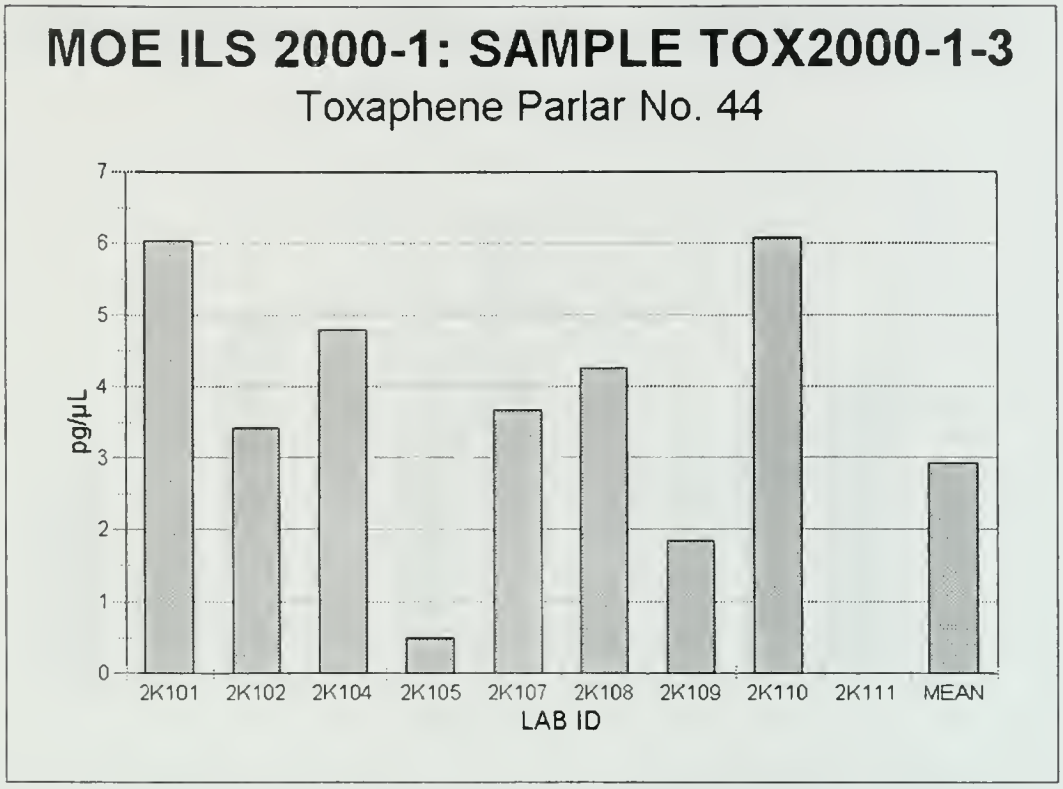

Figure 5 - Parlar 44 in Sample TOX2000-1-3

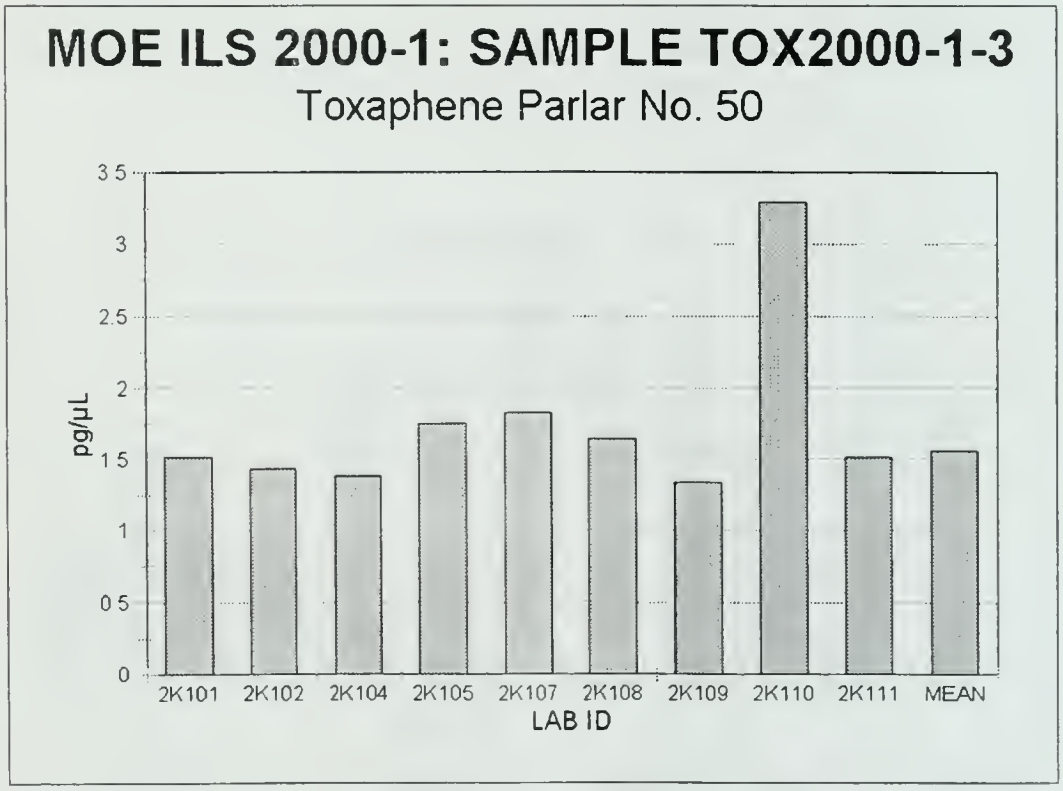

Figure 6 - Parlar 50 in Sample TOX2000-1-3

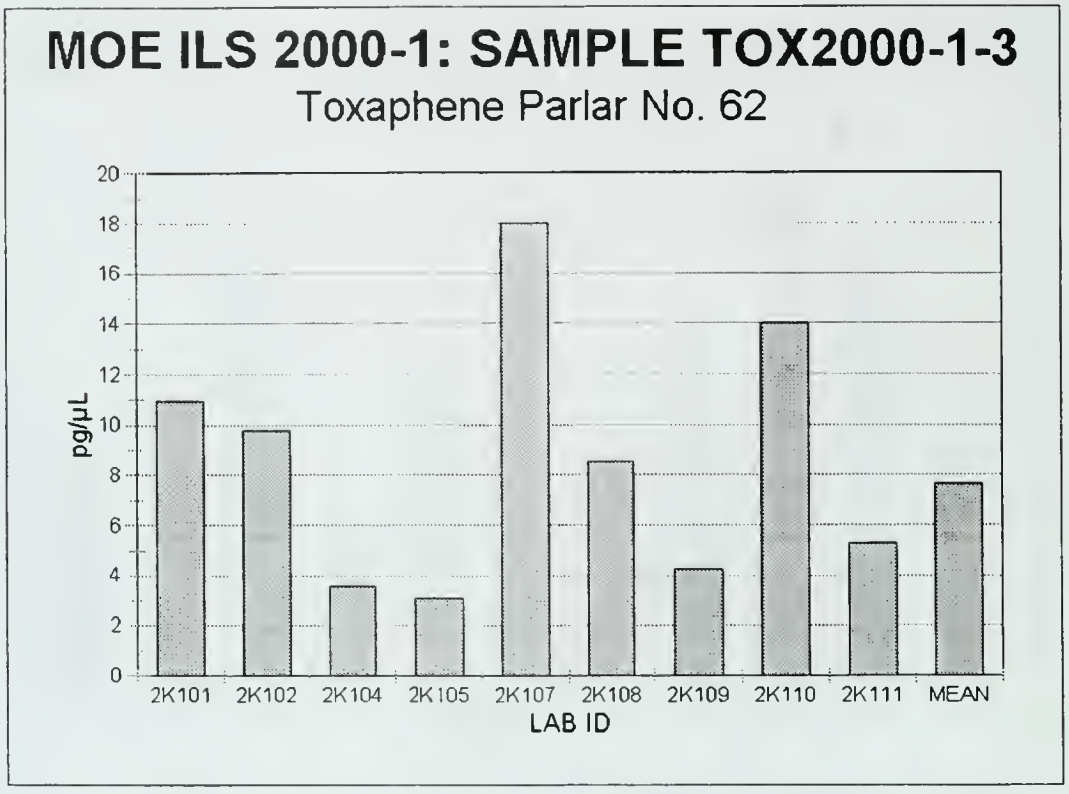

Figure 7 - Parlar 62 in Sample TOX2000-1-3

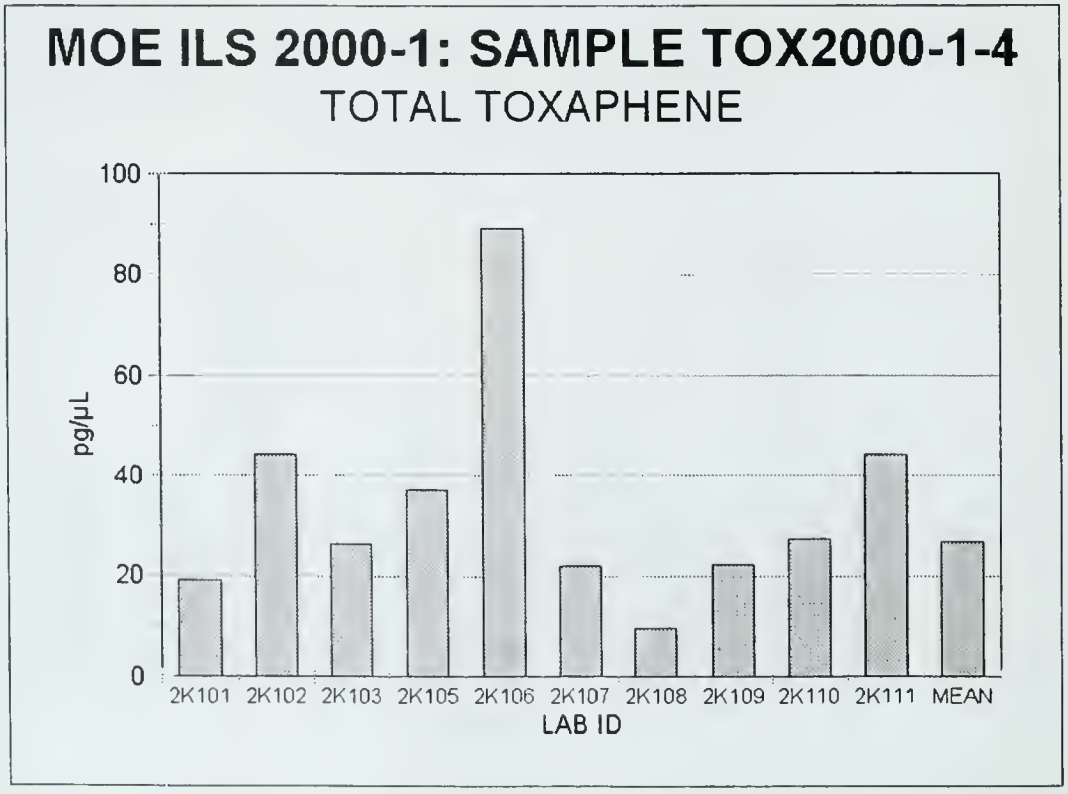

Figure 8 - Total Toxaphene in Sample TOX2000-1-4

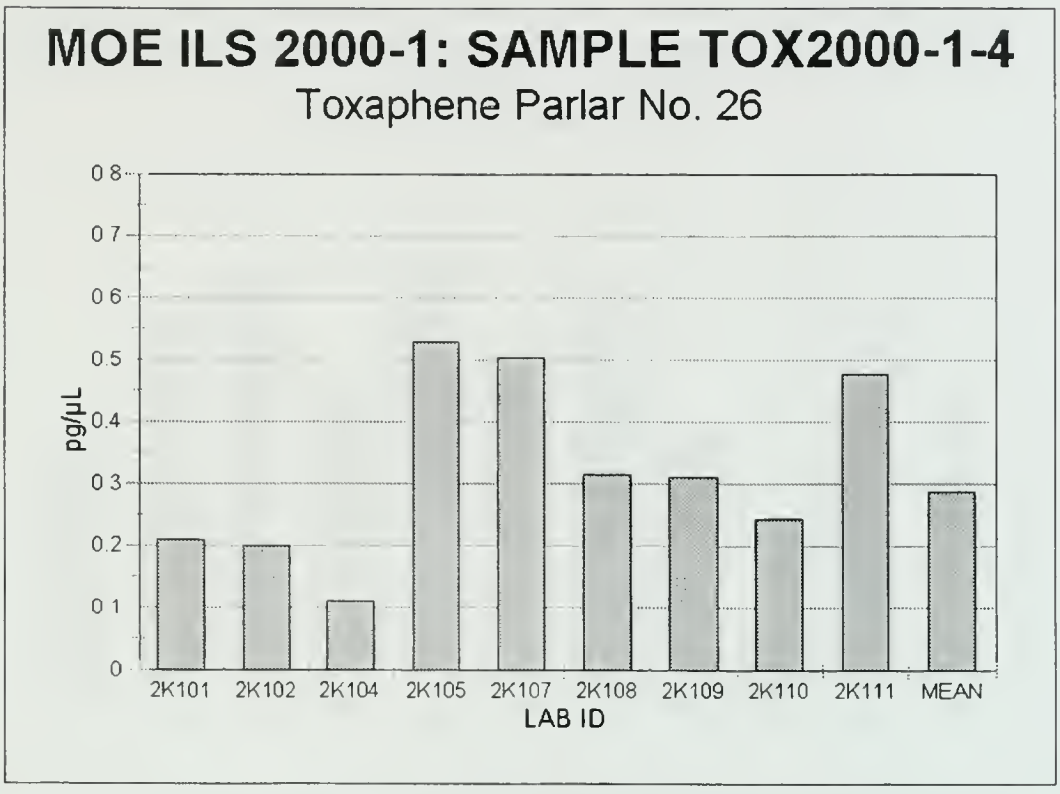

Figure 9 - Parlar 26 in Sample TOX2000-1-4

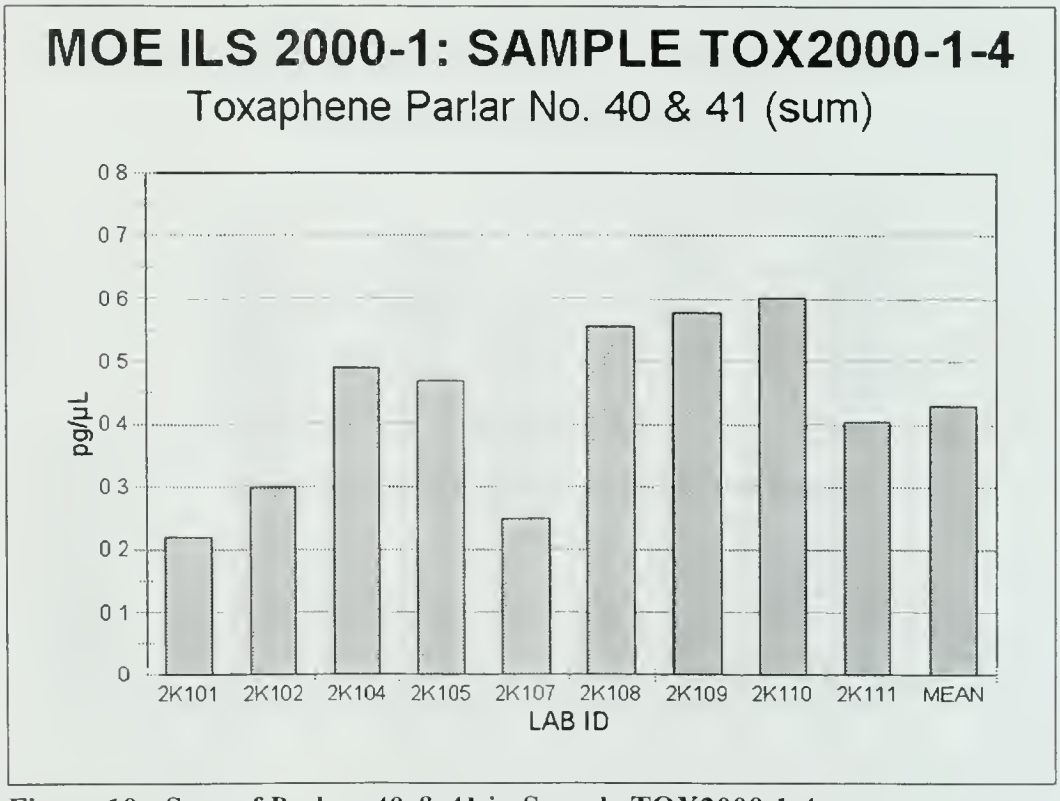

Figure 10 - Sum of Parlars 40 & 41 in Sample TOX2000-1-4

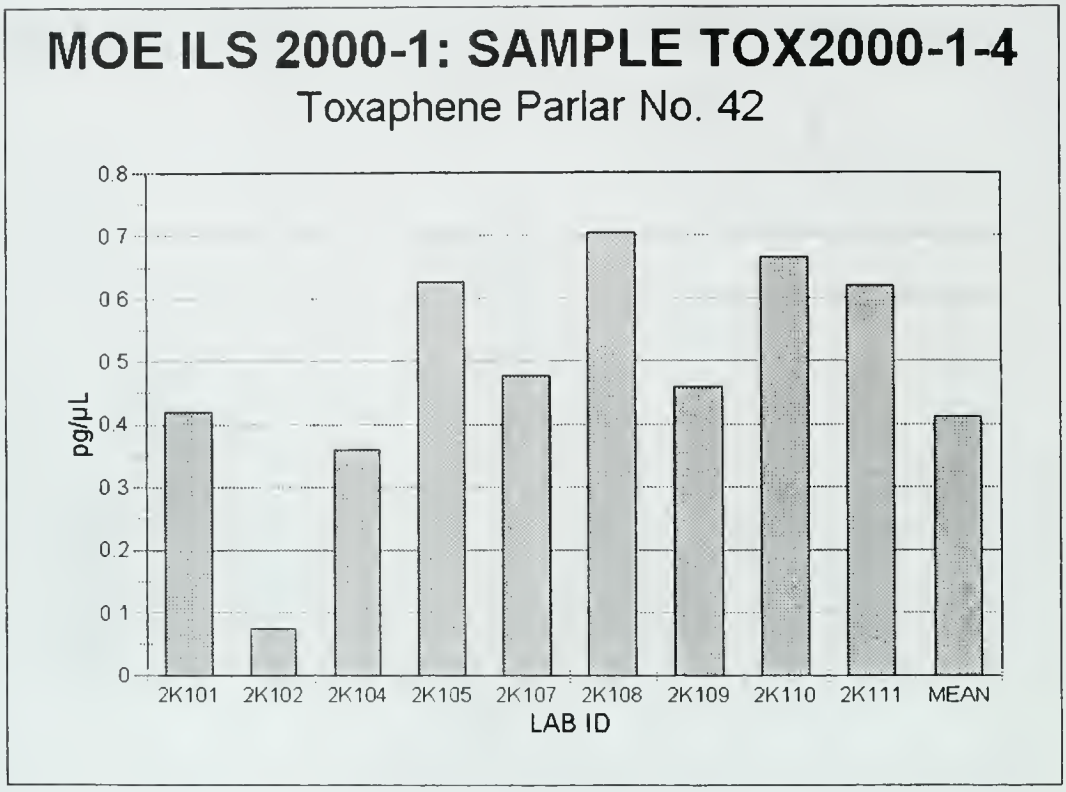

Figure 11 Parlar 42 in Sample TOX2000-1-4

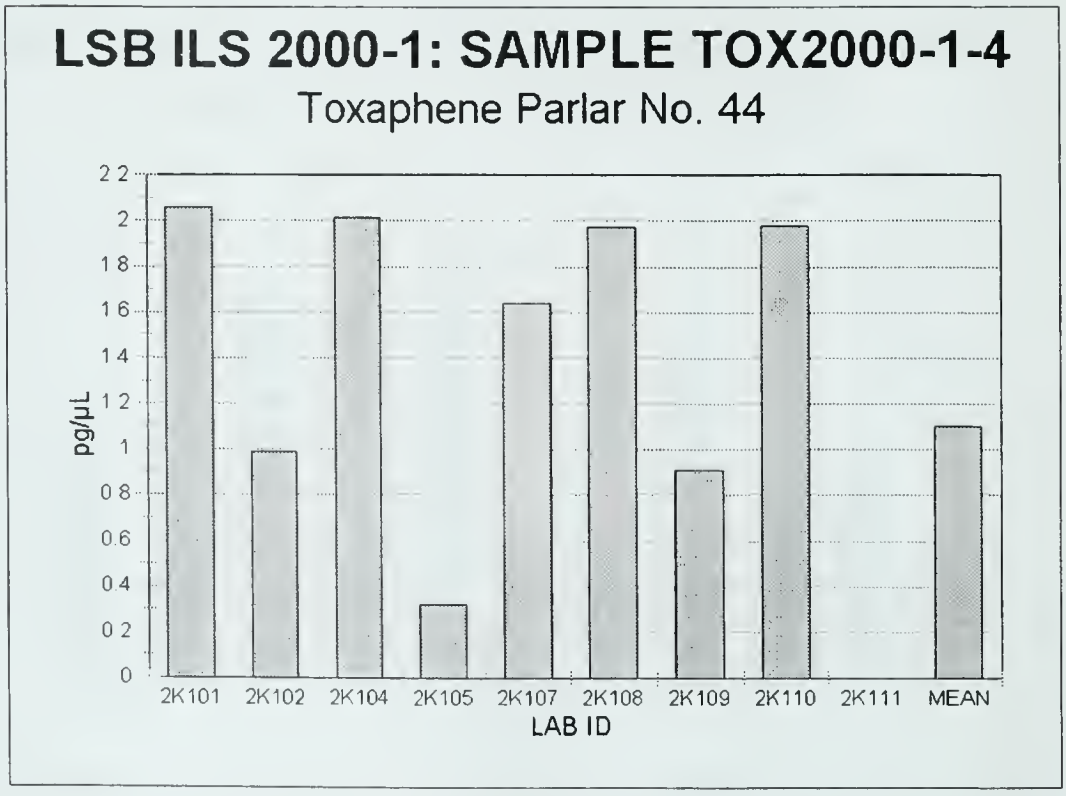

Figure 12 - Parlar 44 in Sample TOX2000-1-4

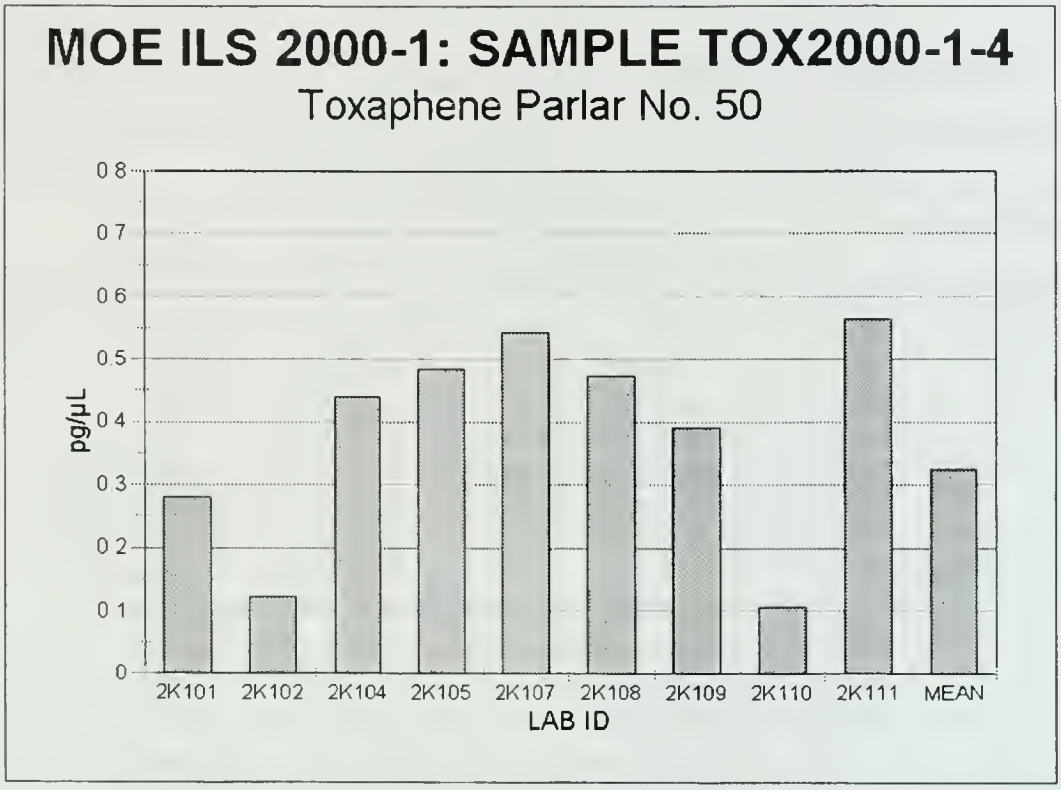

Figure 13 - Parlar 50 in Sample TOX2000-1-4

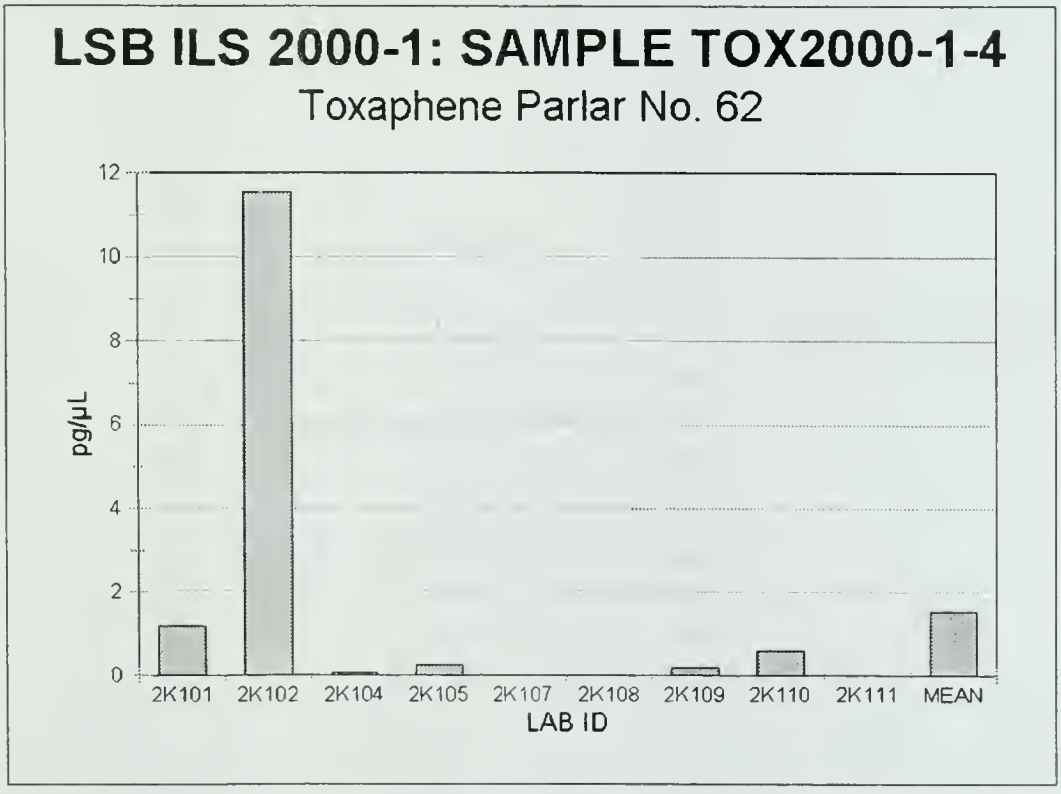

Figure 14 - Parlar 62 in Sample TOX2000-1-4

9. APPENDIX 3 - METHODOLOGY SUMMARY

TABLE 4 - Methodology Summary		
Sample Storage	Refrigerator Freezer	4 labs 7 labs
Surrogate	No ^{13}C-Chlordane α-HCH ^{13}C-PCB118 PCB30 & PCB204 $^{13}C_{12}$-PCB180	6 labs 1 lab 1 lab 1 lab 1 lab 1 lab
Clean-up Column	Florisil Deactivated silica Activated silica Silica & alumina HP5-MS no information	2 labs 1 lab 3 labs 1 lab 1 lab 3 labs
Final solvent	Hexane Isooctane	6 labs 5 labs
Final sample volume	TOX2000-1-3 TOX2000-1-4	50 µL to 1.1 mL 50 µL to 5.5 mL
GC Column	DB-5, 30 m DB5-MS, 30 m DB-5, 60 m DB5-MS, 60 m DB-XLB, 30 m HP5-MS HP Ultra 2, 25 m & HP-1, 15 m MDN-5, 30 m	1 lab 1 lab 2 labs 3 labs 1 lab 1 lab 1 lab 1 lab
GC Temperature Program	Start 80°C; End 290°C Start 80°C; End 300°C Start 40°C; End 300°C Start 80°C; End 315°C Start 90°C; End 280°C Start 120°C; End 280°C Start 80°C; End 285°C Start 80°C; End 265°C Start 90°C; End 270°C Start 80°C; End 280°C	1 lab 2 labs 1 lab 1 lab 1 lab 1 lab 1 lab 1 lab 1 lab 1 lab
Carrier Gas	Helium	11 labs

TABLE 4 - Methodology Summary		
Instrument	GC/ECNI	8 labs
	GC/LRMS	1 lab
	GC/ECNI & GC/LRMS	1 lab
	GC/ECNI, GC/LRMS & GC/HRMS	1 lab
Calibration	Single point	5 labs
	2 points	1 lab
	Multi-point	2 labs
	Multi-point, continuous	3 labs
Labeled Standard	None	2 labs
	$^{13}C_8$-Mirex	1 lab
	PCB204	4 labs
	$^{13}C_{12}$-PCB153	1 lab
	2,3,6,7-tetrachloronaphthalene	1 lab
	$^{13}C_{10}$ t-nonachlor	1 lab
	Octachloronaphthalene & 1,3,5-tribromobenzene	1 lab
Injection Volume	1 µL	3 labs
	2 µL	7 labs
	2 mL	1 lab
Correct for Recovery	Yes	7 labs
	No	4 labs

TABLE 4 - Methodology Summary		
Calculation of "Total" Toxaphene	2K101	Sum hexa to nona homologue concentrations
	2K102	Correct for ^{13}C & chlordane interferences; sum hexa through deca homologues
	2K103	Correct for ^{13}C & chlordane interferences; sum hexa through deca homologues
	2K104	Not calculated
	2K105	Sum areas m/z 377, 379, 381, 411, 413, 415 (Cl_8 & Cl_9 bornane homologues)
	2K106	Check ratios of quantitation/confirmation ions; sum quantitation ions for hexa to deca homologue groups
	2K107	Sum of ions 342 (hexa), 343 (hepta), 377 (octa), 413 (nona), 449 (deca); correct for chlordane, C^{19} & β-endosulfan interferences
	2K108	Sum of 40 "assignments" of peaks and/or "clusters" that cover range of hexa to deca homologues
	2K109	Monitor ions 343, 345 (7-Cl), 379, 381 (8-Cl), 413, 415 (9-Cl); sum total peak areas of the 343, 379 & 413 ions
	2K110	Sum of ions 307 (hexa), 343 (hepta), 377 (octa) & 411 (nona); correct for chlordane & endosulfan inferences
	2K111	Sum of hexa to deca chlorinated bornanes/bornenes
Laboratory Accredited	Yes	2 labs
	No	9 labs
PE Program Participation	Yes	6 labs
	No	5 labs

10. APPENDIX 4 - LIST OF PARTICIPANTS

Atmospheric Contaminant Impacts, National Water Research Institute, Environment Canada; Burlington, Ontario, Canada

Axys Analytical; Sidney, British Colombia, Canada

Chesapeake Biological Laboratory; Solomons, Maryland, U.S.A.

Environmental Chemistry Laboratories, University of Minnesota; Minneapolis, Minnesota, U.S.A.

Freshwater Institute, Department of Fisheries and Oceans Canada; Winnipeg, Manitoba, Canada

Hazardous Air Pollutants Laboratory, Meteorological Service Canada, Environment Canada; Downsview, Ontario, Canada

Hites Laboratory, Indiana University; Bloomington, Indiana, U.S.A.

MSC Organics Analysis Laboratory, Meteorological Service Canada, Environment Canada; Downsview, Ontario, Canada

National Laboratory for Environmental Testing, National Water Research Institute, Environment Canada; Burlington, Ontario, Canada

Norwegian Institute for Air Research; Kjeller, Norway

Skidaway Institute of Oceanography; Savannah, Georgia, U.S.A.

11. APPENDIX 5 - CORRESPONDENCE

ONTARIO MINISTRY OF THE ENVIRONMENT
INTERLABORATORY STUDY 2000-1

TOXAPHENE

STANDARDS AND AIR SAMPLE EXTRACT

LABORATORY INFORMATION QUESTIONNAIRE

The Quality Management Unit of the Ontario Ministry of the Environment, Laboratory Services Branch is conducting an interlaboratory study on behalf of the Atmospheric Environment Service (AES), Environment Canada. You have been previously contacted about participating in this study by Terry Bidleman of AES. This study supports the Integrated Atmospheric Deposition Program (IADN) as well as other air programs. The target analytes are Total Toxaphene and selected Toxaphene congeners of environmental concern. This study will consist of a series of injection ready standards for instrumental analysis, plus one (1) air sample extract to be processed through analytical clean-up before instrumental analysis. The samples will be distributed at the end of February 2000, with 3 months allowed to report results.

To ensure that the study design is appropriate for the study coordinators and the participating laboratories, the following information is requested from laboratories interested in participating in this study.

Laboratory Name:

Contact Person:

Telephone:

FAX:

e-mail:

Shipping Address:

Matrices Routinely analyzed for Toxaphene:

Please complete the following table as it applies to your laboratory. The Toxphene congeners listed in the table are those proposed as target analytes for this study. If you routinely analyze for other congeners, please add them to the blank spaces.

COMPOUND	ROUTINELY ANALYZED (Y/N)	FULL-SCALE (INSTRUMENTAL)	MDL	UNITS
Total Toxaphene				
P26 2-endo-, 3-exo-, 5-endo-, 6-exo-, 8, 8, 10, 10- Octachlorobornane				
P32 2, 2, 5-endo, 6-exo, 8, 9, 10- Heptachlorobornane				
P40 2-endo, 3-exo-,5-endo-, 6-exo-, 8, 9, 10, 10- Octachlorobornane				
P41 2-exo, 3-endo, 5-exo, 8, 9, 9, 10, 10- Octachlorobornane				
P42 2, 2, 5-endo, 6-exo, 8, 8, 9, 10- Octachlorobornane				
P44 2-exo, 5, 5, 8, 9, 9, 10, 10- Octachlorobornane				
P50 2-endo-, 3-exo-, 5-endo-, 6-exo-, 8, 8, 9, 10, 10- Nonachlorobornane				

Please return this questionnaire by **December 17, 1999** to:

Sylvia Cussion
ILS Coordinator
FAX: +1 416 235 6312
e-mail: cussiosy@ene.gov.on.ca

Quality Management Unit

Contact
Laboratory
Address
Country

April 4, 2000

RE: MOE INTERLABORATORY STUDY 2000-1
TOXAPHENE: STANDARDS & AIR SAMPLE EXTRACT

Dear Contact,

Please find enclosed the following samples for Interlaboratory Study (ILS) 2000-1:

Sample TOX2000-1-1 - Ampouled Toxaphene Congener Standard in Iso-Octane
Sample TOX2000-1-2 - Ampouled Toxaphene Parlar 42 Standard in Iso-Octane
Sample TOX2000-1-3 - Ampouled Toxaphene Solution (Iso-Octane)
Sample TOX2000-1-4 - Ampouled Air Extract in Iso-Octane

Please contact me if you did not receive all of the samples intact and we will send replacement samples.

The samples are to be analyzed using your routine methods for Toxaphene, both Total and specific Congeners. **Please read the enclosed instruction sheet before proceeding with the analysis.**

Enclosed is a report form for the results and a questionnaire regarding your methodology to be returned with your results. The report form and questionnaire will also be provided to you electronically by e-mail. This will be in Excel and Word (Rich Text Format). Please report your results electronically, if possible.

Please return all results by **May 31, 2000**.

Your confidential study ID Code is: ID Code

Thank you for participating in this interlaboratory study.

Sylvia Cussion
ILS Coordinator
+1 416 235 6348
FAX: +1 416 235 6312
e-mail: cussiosy@ene.gov.on.ca

MOE INTERLABORATORY STUDY 2000-1
TOXAPHENE STANDARDS & AIR SAMPLE EXTRACT

SAMPLE INSTRUCTION SHEET

SAMPLES TOX2000-1-1 & TOX2000-1-2: TOXAPHENE CONGENER CALIBRATION STANDARDS

These two solutions are in Iso-Octane and are ready for direct instrumental injection. They are provided as calibration solutions for specific Toxaphene Congener Analysis.

Sample TOX2000-1-1 contains the following individual congeners: Parlar No. 26, Parlar No. 40, Parlar No. 41, Parlar No. 44, Parlar No. 50, and Parlar No. 62. All are at a concentration of **30 pg/µL**.

Sample TOX2000-1-2 contains one congener, Parlar 42, at a concentration of **34 pg/µL**.

SAMPLE TOX2000-1-3: AMPOULED TOXAPHENE SOLUTION

This is an unknown Toxaphene solution in Iso-Octane. The expected concentration range is 100-200 pg/µL. Please use your routine Toxaphene standard(s) to calibrate your instrument and report a "Total" Toxaphene result. Please use solutions TOX2000-1-1 and TOX2000-1-2 as calibration standards for the individual congeners. If you repeat the analysis with your own congener calibration solution and wish to report duplicate results, please do so, and identify the source and concentration of your standards. **Please report all of your results with a minimum of 3 figures.**

SAMPLE TOX2000-1-4: AIR SAMPLE EXTRACT

Please analyze this sample using your routine method.

Please use your routine Toxaphene standard(s) to calibrate your instrument and report a "Total" Toxaphene result. Please use solutions TOX2000-1-1 and TOX2000-1-2 as calibration standards for the individual congeners. If you repeat the analysis with your own congener calibration solution and wish to report duplicate results, please do so, and identify the source and concentration of your standards. **Please report all of your results with a minimum of 3 figures.**

REPORTING RESULTS

A hard copy of the results report form and the methodology questionnaire have been provided. Electronic copies will be provided by e-mail. Please report the results electronically by e-mail (cussiosy@ene.gov.on.ca or selliasa@ene.gov.onb.ca). The results report form will be provided in Excel and the methodology questionnaire in Word (Rich Text Format).

To confirm the accuracy of the electronic transmission of results, please forward a hard copy of the results, either by FAX (+1 416 235 6312) or mail.

ONTARIO MINISTRY OF THE ENVIRONMENT
INTERLABORATORY STUDY 2000-1
TOXAPHENE STANDARDS & AIR SAMPLE EXTRACT
APRIL 2000

DUE DATE: MAY 31, 2000

LABORATORY ID CODE:

1. SAMPLE RECEIPT

Date Samples Received: _____

Date Analysis Initiated: _____

Storage Condition of Samples Prior to Analysis: _____

_____

2. SAMPLE CLEAN-UP

Are "Clean-up" Standards added to the sample: Yes [ ]No [ ]

If "yes", please identify type, source, and when they are added to the sample:_____

_____

Column:

Other Column(s):

Other Procedure (eg. adsorbant in a beaker):

Final Solvent of Sample: _____

Final Volume of Sample: _____

4. INSTRUMENTAL ANALYSIS

GC Column: _____

GC Program: _____

ONTARIO MINISTRY OF THE ENVIRONMENT
INTERLABORATORY STUDY 2000-1
TOXAPHENE STANDARDS & AIR SAMPLE EXTRACT
APRIL 2000

DUE DATE: MAY 31, 2000

LABORATORY ID CODE:

Head Pressure:_____ **Carrier Gas:** _____

Please circle the following which applies: GC/LRMS GC/HRMS GC/MS/MS

GC/ECNI

Manufacturer and Model: _____

_____

Resolution: _____

Source of Calibration Standards: _____

Type of Calibration: single-point [ ] two-point [ ] multi-point [ ]

multi-point with continuing calibration [ ]

Addition of further labeled standard to extract immediately prior to instrumental analysis:

Yes [ ] No [ ]

If "Yes", source and type of standards used: _____

_____

Volume of sample injected: _____

Do you routinely correct sample results for recovery of surrogates? Yes [ ]No [ ]

Please describe how you quantify for "Total" Toxaphene. What homolgues are summed?

ONTARIO MINISTRY OF THE ENVIRONMENT
INTERLABORATORY STUDY 2000-1
TOXAPHENE STANDARDS & AIR SAMPLE EXTRACT
APRIL 2000

DUE DATE: MAY 31, 2000

LABORATORY ID CODE:

5. LABORATORY INFORMATION

Is your laboratory accredited for this analysis Yes [ ]No [ ]

If "yes", who is the accreditation body and standard used? _____

_____

Does your laboratory participate in a proficiency testing program for this analysis?

Yes [ ]No [ ]

If "yes", please identify the program. _____

_____

How many years has your laboratory performed analysis for toxaphene? _____

Approximately how many samples of this type do you analyze annually? _____

General Comments re ILS 2000-1

Quality Management Unit

Contact
Laboratory
Address
Country

November 24, 2000

RE: MOE INTERLABORATORY STUDY 2000-1
TOXAPHENE: STANDARDS & AIR SAMPLE EXTRACT

Dear Contact,

Please find enclosed the preliminary tables of results for Interlaboratory Study 2000-1. Please check that there were no data transcription errors and report any corrections no later than **December 22, 2000**.

I apologize for the delay in providing this information to you. I was asked to delay reporting back to the participants in the hope that the final participant would be able to report results.

A final report will be written and should be available early in 2001.

Your confidential study ID Code is: ID Code

Thank you for participating in this interlaboratory study.

Sylvia Cussion
ILS Coordinator
+1 416 235 6348
FAX: +1 416 235 6312
e-mail: cussiosy@ene.gov.on.ca